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THE EFFECT OF
CARBON AND BORON ON THE
FRICTION BEHAVIOR OF
TI-IMPLANTED 52100 BEARING STEEL
AT LOW SLIDING SPEEDS

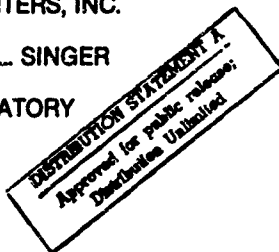
PREPARED FOR
THE U.S. NAVAL RESEARCH LABORATORY
UNDER CONTRACT NUMBER
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320 NEEDHAM STREET
NEWTON UPPER FALLS, MASSACHUSETTS 02164

AUTHORED BY
RUSSELL A. JEFFRIES OF GEO-CENTERS, INC.
AND
ROBERT N. BOLSTER AND IRWIN L. SINGER
OF THE
U.S. NAVAL RESEARCH LABORATORY

MAY 1982



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PREFACE

Although the work summarized in this report was performed by Geo-Centers, Inc., Mr. Bolster and Dr. Singer of the U.S. Naval Research Laboratory have been included as authors in acknowledgment of their valuable discussions on the theoretical implications of the program.

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ACKNOWLEDGMENTS

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ABSTRACT

↓
The low speed friction behavior of Ti implanted AISI 52100 bearing steel (1.5% Cr, 1% C by weight) may be altered by varying the concentration and distribution of the titanium and carbon present in the surface. With the aid of carbon which is either directly implanted or gettered during the implantation process, wear resistant, low friction surfaces may be produced. By assuring a sufficient supply of carbon, the Ti dosage required to produce a wear resistant surface may be reduced by more than 50%. Transmission electron microscopy of dual implants of carbon and titanium each at a dose of 2×10^{17} ions/cm² showed that the surface was still crystalline and typical for a martensitic material. In contrast, friction testing of samples implanted with titanium and boron, another interstitial species forming wear resistant species (i.e., TiB₂), did not produce surfaces which were wear resistant nor substantially reduce the friction. Auger analysis in conjunction with ion milling was used to compare compositions of implanted surfaces. ↗

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1. INTRODUCTION

The implantation of various ion species and their effects on tribological behavior are well documented(1,2,3). The implantation of titanium into AISI 52100 bearing steel has been shown to have a marked influence on the coefficient of friction, abrasive wear resistance, and microstructure of the steel(3,4). The effect of the implantation on the aforementioned properties was strongly dependent on the amount of titanium implanted. A model for the gettering of carbon by the exposed surface titanium has been proposed by Singer et al.(3); the presence of carbon was responsible for the improvements seen in titanium implanted steel. In this program, the implantation of titanium and the effects of carbon and boron on the tribological properties are studied for several cases:

- 1) the implantation of titanium at high energy into samples which have already been implanted with carbon;
- 2) the implantation of carbon and titanium at a fixed dose with varying orders of implantation;
- 3) the implantation of titanium at low energy; and
- 4) the dual implantation of titanium and boron at high energy.

Friction measurements and wear scar analysis were used to evaluate the effect of the surface modification. Ion milling and Auger analysis provided compositional analysis and chemical information on the implanted surfaces.

2. EXPERIMENTAL PROCEDURES AND DATA ANALYSIS

2.1 IMPLANTATION

Implantation of AISI 52100 bearing steel samples was done by using a modified Varian/Extrion Model 200-20A2F ion implanter. A hot filament (cathode) arc discharge-type ion source was used. In this process, titanium chlorides are produced by passing chlorine gas through a fine titanium powder contained in a graphite chamber. At the high source temperature (800°C), the chlorides are volatilized and ionized. The intensity of the resulting beam ($^{48}\text{Ti}^+$) is approximately 200 to 800 μA . Gaseous CO was used for a carbon source; the boron source was boron trifluoride.

The 52100 samples were disks 0.95 cm in diameter and 0.3 cm thick. During implantation, these samples were heat-sunk on a water-cooled holder and were kept at temperatures less than 40°C. The target chamber was maintained at pressures of approximately 5×10^{-7} torr with cryogenic pumps. Ion beams were electrostatically raster scanned over the sample holder to give averaged uniform current densities of about 15 $\mu\text{A}/\text{cm}^2$.

The dual implantation at various Ti doses was carried out by implanting three disks with $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV, and subsequently with $5 \times 10^{16} \text{ Ti}^+/\text{cm}^2$ at 190 keV. One of the disks was then removed, and the other two disks placed back in the chamber and implanted until the cumulative dose was $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$. One of these two disks was then removed and the procedure was repeated with the remaining disk, which received $5 \times 10^{17} \text{ Ti}^+/\text{cm}^2$.

The next dual implant series was done at a constant dose of 2×10^{17} ions/ cm^2 , and involved three sets of two disks each. One of each pair of disks had two transmission electron microscopy (TEM) disks mounted on its face. The first pair was implanted with $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV. All three

pairs were then implanted at the same time with $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV, and the first and second pairs were then removed. The last pair was implanted subsequently with $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV.

The low energy implantation of titanium was carried out at 55 keV by implanting three disks to $5 \times 10^{16} \text{ Ti}^+/\text{cm}^2$, removing one disk and continuing until cumulative doses were reached at $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ and $5 \times 10^{17} \text{ Ti}^+/\text{cm}^2$, with a disk removed at $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$.

Dual implants with boron were prepared by implanting a disk to $2 \times 10^{17} \text{ B}^+/\text{cm}^2$ and another to $3 \times 10^{17} \text{ B}^+/\text{cm}^2$ at an energy of 40 keV. The two disks were subsequently implanted simultaneously to a dose of $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV.

2.2 FRICTION MEASUREMENTS

Kinetic coefficient of friction measurements were done on a "stick-slip" machine. The slider of 52100 steel was a 1.27 cm diameter sphere which had been cleaned in refluxing benzene in a Soxhlet extractor. The spheres had a surface roughness of $0.025 \mu\text{m}$. The slider was attached to an elastically restrained friction arm, and disks were mounted on a steel bar and clamped to a sliding pad so that the disk moved under the steel slider. Two pairs of resistance strain gauges bonded to the arm measured the normal and tangential forces.

Prior to implantation, the disks were prepared by grinding on successively finer abrasive papers (through 3/0 emery) using water or kerosene/stearate as a lubricant. Rough polishing was done with $3 \mu\text{m}$ diamond to a mirror finish; fine polishing was done using $0.05 \mu\text{m}$ γ -alumina. After fine polishing, the samples were lightly etched with 1% Nitral to ensure that any surface damage was removed, and then repolished with a $0.05 \mu\text{m}$ γ -alumina. The bulk hardness of the sliders and the disks was about $R_C = 60$. Before measurements were made, the clean samples were rinsed again with acetone and then 2-propanol.

The kinetic coefficient of friction (μ_k) was measured in air at $\sim 23^\circ\text{C}$ at a sliding velocity of 0.01 cm/sec . No lubricant was normally used. A normal force of 9.8 N was used, resulting in a peak Hertzian pressure of

0.57 GPa (83,000 psi). Multiple unilateral traverses (passes) were made on the surface, the first pass being 5 mm in length and subsequent passes 3 mm in length. The first tests were to determine the friction values until the friction values returned to the original steel-on-steel values. The slider was then changed, the sample was moved, and the measurements were repeated to the point at which the changes occurring in the first test were reached. The tracks made during friction testing were photographed with a Michelson interference microscope using monochromatic light (yellow) with a fringe spacing of 294 nm.

2.3 AUGER ANALYSIS

Auger analysis was performed in a UHV chamber equipped with a Perkin-Elmer (PHI) Model 545 Auger microprobe, a rasterable ion gun, Ti sublimators, and liquid nitrogen cooled cryopanel. The electron gun was operated at 2 kV with a 2 μ A beam rastered over a spot, 50 μ m in diameter. Derivative spectra were recorded directly at a 3 eV modulation amplitude. The ion gun was operated in an Ar atmosphere (5×10^{-5} torr) with a rastered beam of 2 keV Ar^+ ions at densities ranging from 2 to 25 $\mu\text{A}/\text{cm}^2$. Composition vs depth profiles were done with a peak-height recording multiplexer operating at a 3 eV modulation amplitude in conjunction with ion milling with Ti sublimators in operation and the cryopanel at LN_2 temperature. Quantitative analysis was performed with methods previously described(3). Table 1 is a summation of the sensitivity factors used.

Table 1. Sensitivity factors for analysis at a modulation amplitude of 3 eV.

Auger Peak	B ₁₈₀	Ar ₂₁₅	C ₂₇₀	Ti ₄₂₀	O ₅₁₀	Cr ₅₃₀	Fe ₆₅₀
Sensitivity	0.15	1.0	0.47	0.48	0.5	0.36	0.17

Depth profiles were obtained for samples by masking a small area on the unworn surface and, once profiling was completed, removing the mask and measuring the crater depth with the interferometer. Intermediate depths were taken to be proportional to the ion milling time. Line spectra were obtained

at selected places during the profile. The sample was then remasked to outline a portion of the wear scar which was left at the point the friction changed.

2.4 TRANSMISSION ELECTRON MICROSCOPY

Specimens were prepared by cutting a 250 μm thick slice from a 52100 disk with a low-speed diamond saw. These slices were: 1) mounted and parallel-ground with 3/0 emery on both sides to 125 μm ; 2) spark-machined to give three 3 mm disks, which were remounted and polished to the same finish on one side as the friction disks; 3) mounted on a friction disk; and 4) implanted on the polished side according to the procedure previously described. After implantation, the TEM disks thinned from the back using a dual-jet electropolisher and an electrolyte of 250 ml methyl alcohol, 150 ml n-butyl alcohol, and 30 ml perchloric acid at a voltage of 90 V, current of 16 mA, and a temperature of 213 K. The samples were rinsed twice each in methanol and ethanol.

Microscopy and selected area electron diffraction patterns were done on a JEM-200CX equipped with an X-ray emission spectrometer (XES) and an electron energy loss spectrometer (EELS).

3. RESULTS AND DISCUSSION

The dual implantation of carbon and titanium was an improvement over samples implanted with titanium only. Figure 1 shows the effect of implantation of carbon and, subsequently, with titanium to three different doses.

Some improvement in the friction behavior was found for the 5×10^{16} Ti^+/cm^2 dose when compared to a similar sample lacking the implanted carbon, but the most dramatic change in behavior was found for the 2×10^{17} Ti^+/cm^2 dose. In a sample which had only the titanium implanted, the friction coefficient had low average values over the first few passes, but rose to a value of $\mu_k \sim 0.9$ after about 10 passes. The friction then dropped slowly to the value for a nonimplanted disk at steady state ($\mu_k = 0.62$). At 10 passes, the sample displayed a significant amount (~ 30 nm) of adhesive wear. In contrast, the dual implantation of carbon and titanium showed a significant improvement in terms of friction and wear. The initial coefficient of friction was low for the first few passes, with a slow rise to a value $\mu_k = 0.45$. Stick-slip behavior then occurred and remained throughout the test. At 10 passes, the improvement approached 50% over the titanium implant alone, and approximately 25% over the nonimplanted disk. At the highest dose, 5×10^{17} Ti^+/cm^2 , the addition of the implanted carbon did not appear to have any effect on the friction behavior when compared to a sample receiving only the titanium. The coefficient of friction was reduced by 50% from the value obtained on the nonimplanted disk, and there was no apparent wear.

Figure 2 is a differential interference contrast (Nomarski) photograph of a 20-pass wear scar on a sample implanted first by 2×10^{17} C^+/cm^2 at 50 keV and then 2×10^{17} Ti^+/cm^2 at 190 keV. No adhesion has taken place on this sample, although it may be seen where several abrasive particles have been dragged across the surface.

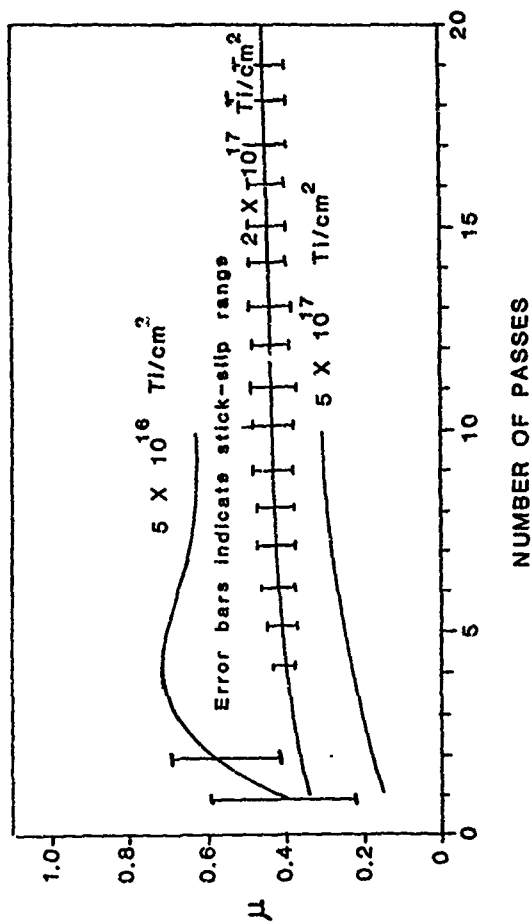


Figure 1. Coefficient of friction vs number of passes for a dual implant of $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 50 keV and three Ti doses: 5×10^{16} , 2×10^{16} , and $5 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV.

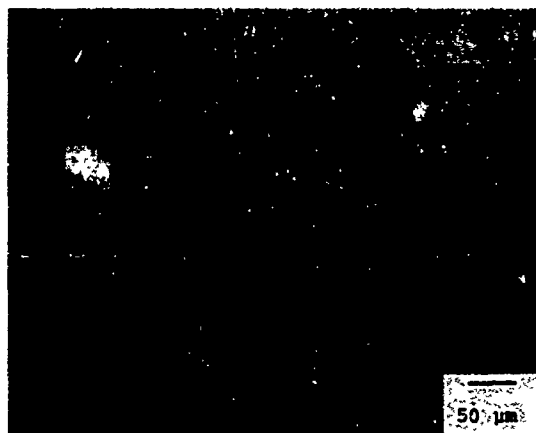


Figure 2. Differential Interference Contrast (DIC or Nomarski) photomicrograph of a dual implant of $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV and $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV after 20 passes.

Table 2 is a summary of interferometer examination of wear scars. As shown in the table, the wear scars produced on samples implanted with carbon and titanium do not show a reduction in wear until a dose of $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ (this sample is No. 3 in the table). The value obtained at these fluences and energy represent a reduction in scar depth by a factor of 3 over the single titanium implantation at the same fluence and energy (No. 9 in the table). All samples possessing $5 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ showed essentially no wear regardless of energy.

The comparison of Auger depth profiles with the data obtained from friction tests and wear scar analysis firmly establishes the necessity that there be significant amounts of carbon and titanium with overlapping profiles located as near to the surface as possible. To best illustrate the effect carbon has on titanium-implanted bearing steel, a dose of $2 \times 10^{17} \text{ ions/cm}^2$ will be compared to doses at $5 \times 10^{16} \text{ ions/cm}^2$ and $5 \times 10^{17} \text{ ions/cm}^2$.

Figure 3 is a depth profile of a sample that had been implanted with $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV and then $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV. At these selected energies, the peak concentration depth was located approximately 70 nm beneath the surface. The concentrations of carbon and titanium at this depth were approximately 20 atomic percent each. Doses of $5 \times 10^{16} \text{ Ti}^+/\text{cm}^2$ and $5 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ resulted in titanium concentrations of ~10 and ~30 atomic percent, respectively. Due to sputtering effects, the depth of the peak Ti concentration moved ~6 nm closer to the surface for each $10^{17} \text{ Ti}^+/\text{cm}^2$ implanted.

Below 50 nm, the Auger lineshape analysis showed that the Ti LMM spectrum and the C KLL spectrum were identical to those encountered by Singer et al.⁽³⁾ In surfaces where carbon was not in sufficient concentration, the Ti LMM spectrum returned quickly to a metal-like form, and the C KLL spectrum changed to produce a doublet which was unlike the spectrum found on the ascending portion of the profile. The dual implanted surface produced a Ti LMM spectrum which remained carbide-like throughout the profile, and the doublet was not formed until the carbon and titanium implants had been completely sputtered through.

Table 2. Interferometry of wear scars.

	Dose (ions/cm ²)	Energy	Number of Passes	Depth (nm)
1.	52100 reference	---	5	SD*
2.	2 x 10 ¹⁷ C ⁺	50	5	SD*
	5 x 10 ¹⁶ Ti ⁺	190		
3.	2 x 10 ¹⁷ C ⁺	50	20	~21
	2 x 10 ¹⁷ Ti ⁺	190		
4.	2 x 10 ¹⁷ C ⁺	50	10	< 5
	5 x 10 ¹⁷ Ti ⁺	190		
5.	2 x 10 ¹⁷ C ⁺	50	15	~20
	2 x 10 ¹⁷ Ti ⁺	190		
6.	2 x 10 ¹⁷ Ti ⁺	190	8	~13
	2 x 10 ¹⁷ C ⁺	50		
7.	2 x 10 ¹⁷ Ti ⁺	190	20	~22
	2 x 10 ¹⁷ C ⁺	50		
8.	2 x 10 ¹⁷ Ti ⁺	190	8	~30
9.	2 x 10 ¹⁷ Ti ⁺	190	20	~60
10.	5 x 10 ¹⁶ Ti ⁺	55	8	~26
11.	5 x 10 ¹⁶ Ti ⁺	55	20	~36
12.	2 x 10 ¹⁷ Ti ⁺	55	20	< 5
13.	5 x 10 ¹⁷ Ti ⁺	55	20	< 5
14.	2 x 10 ¹⁷ B ⁺	40	8	SD*
	2 x 10 ¹⁷ Ti ⁺	190		
15.	3 x 10 ¹⁷ B ⁺	40	8	SD*
	2 x 10 ¹⁷ Ti ⁺	190		

*In cases where SD (severe damage) is indicated, the scars were not measurable because of the rough nature of the damaged surface.

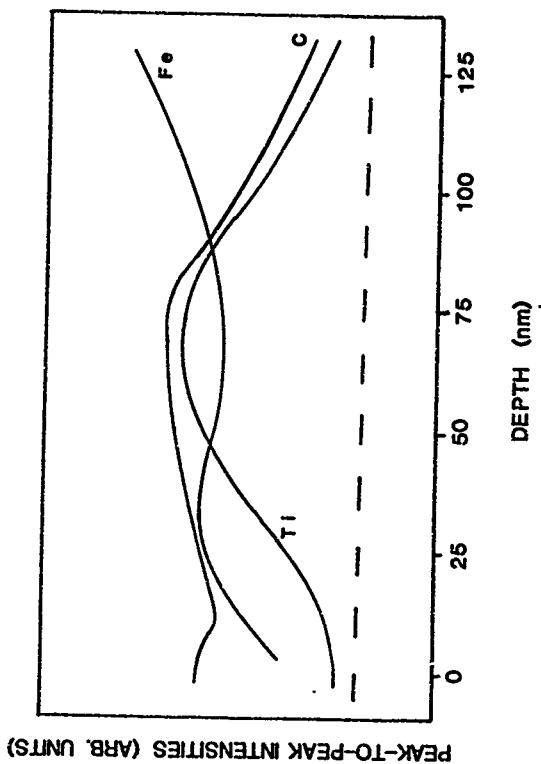


Figure 3. Composition vs depth profile of 52100 steel implanted with $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV and $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV.

Figure 4 shows the friction coefficient for dual implantations of carbon and titanium, each at a dose of 2×10^{17} ions/cm² at energies of 50 and 190 keV, respectively. The order of implantation was of minor consequence at these doses and energies; however, the friction was slightly lower when carbon was the first implant of the series. The average decrease of the friction coefficient at this dose was 20%, with respect to the nonimplanted steady state value. Adhesion was reduced, and wear scars from either of these samples were on the order of 20 nm deep after 20 passes. These were substantial improvements over the nonimplanted case or the single titanium implantation.

Shown in Figure 5 is a TEM micrograph of a sample implanted with 2×10^{17} C⁺/cm² at 50 keV and then 2×10^{17} Ti⁺/cm² at 190 keV. The structure was martensitic, typical for this material, and did not vary in any of the samples examined. The corresponding electron diffraction pattern is shown in Figure 6. To ensure that no titanium and carbon were lost during electropolishing, X-ray Emission Spectroscopy and Electron Energy Loss Spectrometry measurements were performed, and the sample was found to contain 1:1 ratio of carbon to titanium and over 12 atomic percent of each, a nominal value for that dose. There were no remarkable features found during TEM examinations.

The use of 55 keV titanium implantation resulted in lower friction values when compared to any of the previous high energy implantation already discussed. High energy (190 keV) titanium implantation was found to be of little or no value until a dose of 5×10^{17} Ti⁺/cm² was approached. In fact, for doses less than that amount, detrimental effects on friction were observed⁽³⁾. Some improvement was made by using carbon as a dual implant.

Figure 7 shows the coefficient of friction vs the number of unidirectional traverses for the three doses prepared at 55 keV with 5×10^{16} , 2×10^{17} , and 5×10^{17} Ti⁺/cm². In each case, significant improvement was seen compared to a nonimplanted surface. At a dose of 5×10^{17} Ti⁺/cm², it appeared that the low friction value $\mu_k = 0.32$ was obtained regardless of the energy, or the use of additional carbon when compared to other implantations. The friction coefficient at a dose of 2×10^{17} Ti⁺/cm² was lowered further by reducing the energy of the implant. At this dose the friction

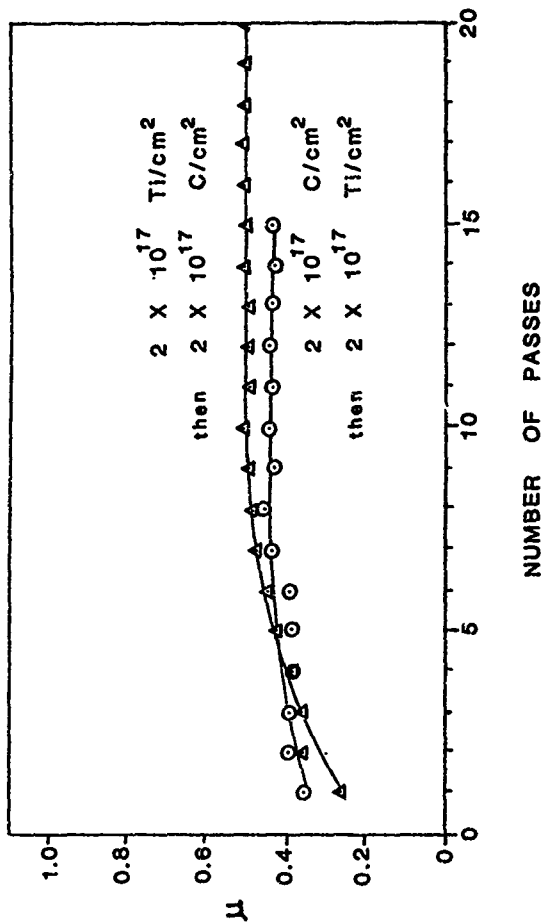


Figure 4. Coefficient of friction vs number of passes for dual implants of 2×10^{17} C⁺/cm² at 50 keV and 2×10^{17} Ti⁺/cm² at 190 keV with implantation order reversed. Stick-slip not shown. Stick slip is ± 0.1 for both samples after 5 passes.



Figure 5. Transmission electron micrograph of $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV and $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV.

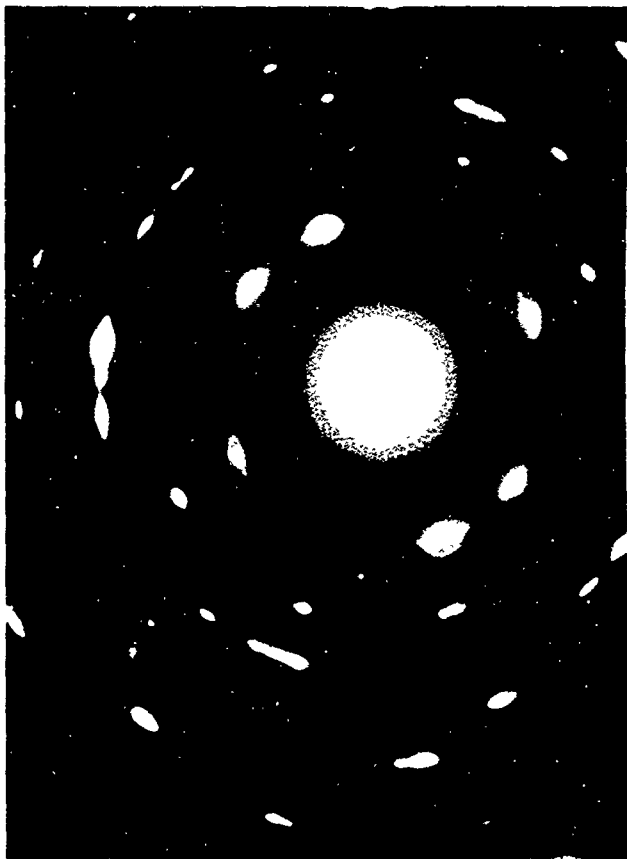


Figure 6. Electron diffraction pattern taken in $2 \times 10^{17} \text{ C}^+/\text{cm}^2$ at 50 keV and $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV.

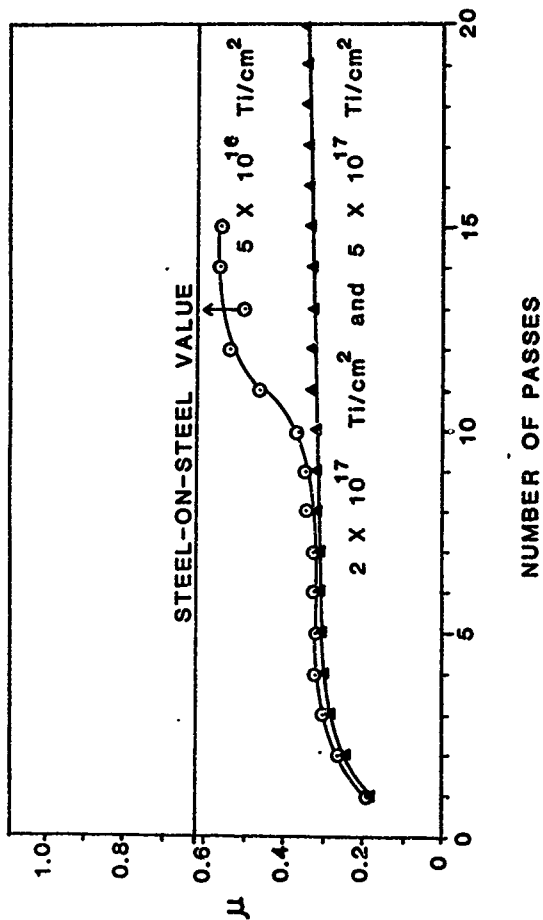


Figure 7. Coefficient of friction vs number of passes for implants of Ti at 5×10^{16} , 2×10^{17} , and 5×10^{17} Ti⁴⁺/cm² at 55 keV.

reached the lowest value obtained ($\mu_k = 0.32$) for any titanium implant in this material and was constant to 20 passes. The wear scar on this sample was not measurable by the interferometer. The friction for the $5 \times 10^{16} \text{ Ti}^+/\text{cm}^2$ was also at this low value for the first 10 passes, and then rose to the steady state value for the nonimplanted steel.

Figure 8 is a Nomarski photo of an 8-pass wear scar on a sample implanted with $5 \times 10^{16} \text{ Ti}^+/\text{cm}^2$ at 55 keV. This scar had begun to show signs of adhesion to the slider, and, at this point, the friction began to rise.

Figure 9 shows the depth profile for a sample implanted with $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 55 keV. The carbon profile overlapped the titanium distribution, with the peak concentration depth $\sim 20 \text{ nm}$ beneath the surface, extending to a maximum of 60 nm. In comparison with $5 \times 10^{17} \text{ Ti}^+/\text{cm}^2$, the highest dose appeared to have a titanium peak concentration at a point which was somewhat deeper than the $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ dose. There also appeared to be separation of the carbon and titanium distributions by $\sim 10 \text{ nm}$. The $5 \times 10^{16} \text{ Ti}^+/\text{cm}^2$ dose was comparable to the $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ dose in terms of its distribution of constituents in the surface.

The dual implantation of B^+ produced surfaces exhibiting initially low coefficients of friction, shown in Figure 10, which rose to the steady state nonimplanted value after relatively short times. In the sample that was richer in boron, some improvement was observed, and the initial stick-slip observed in the sample possessing less boron was eliminated at the higher dose. The adhesion observed was so severe that this surface has little promise as an engineering surface.

Figure 11 is a Nomarski photo of an 8-pass wear scar on a sample implanted with boron and titanium, each at a dose of $2 \times 10^{17} \text{ ions/cm}^2$ at 40 and 190 keV, respectively. The adhesion observed at this point was catastrophic.

Figure 12 shows the depth profile for a sample implanted with $2 \times 10^{17} \text{ B}^+/\text{cm}^2$ at 40 keV and $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV. Peak concentration depth for both species was 75 nm.



Figure 8. Differential Interference Contrast (DIC or Nomarski) photomicrograph of $5 \times 10^{16} \text{ Ti}^+/\text{cm}^2$ at 55 keV after 6 passes.

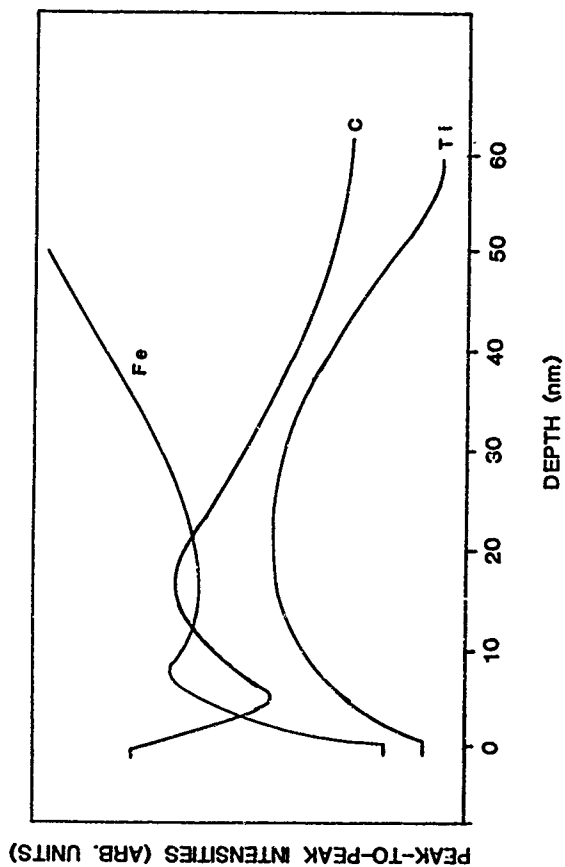


Figure 9. Composition vs depth profile of 52100 steel implanted with $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 55 keV.

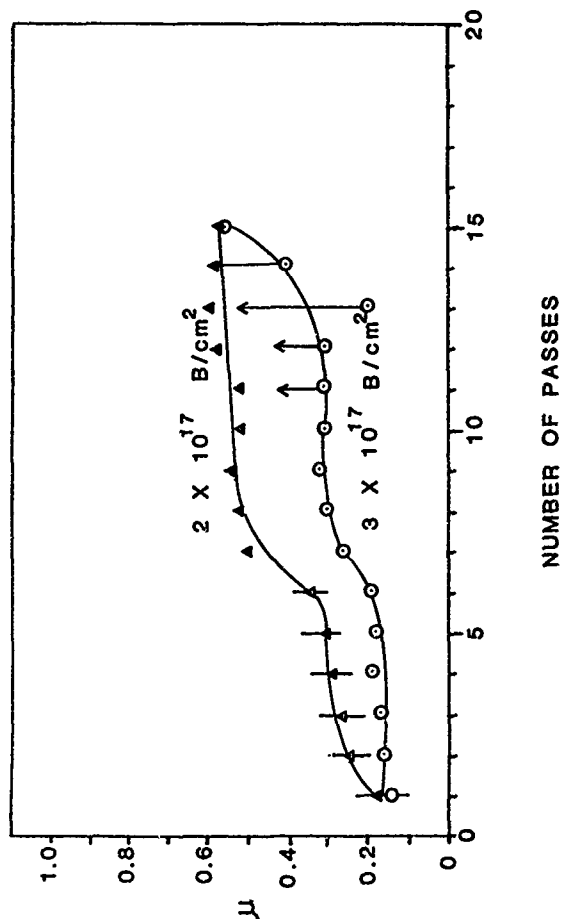


Figure 10. Coefficient of friction vs number of passes for a dual implant of $2 \times 10^{17} \text{ B}^+/\text{cm}^2$ and $3 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV.



Figure 11. Differential Interference Contrast (DIC or Nomarski) photomicrograph of $2 \times 10^{17} \text{ B}^+/\text{cm}^2$ at 40 keV and $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV after 8 passes.

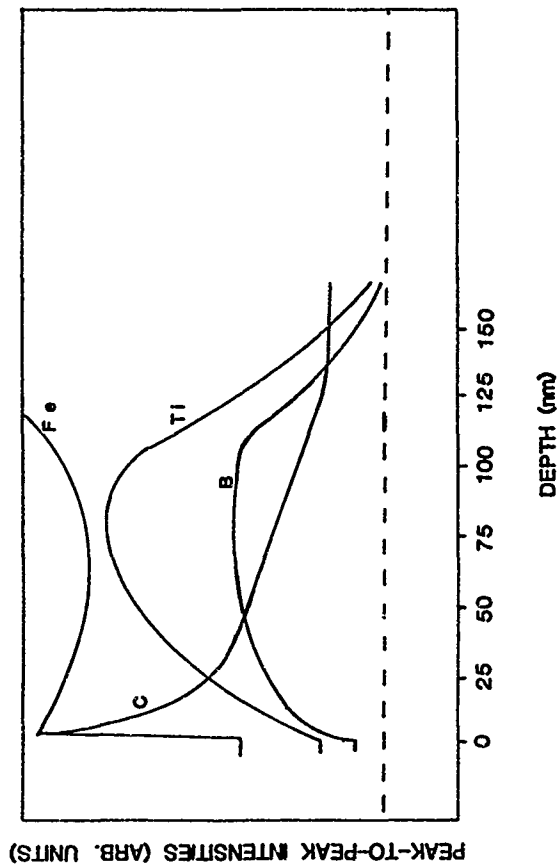


Figure 12. Composition vs depth profile of 52100 steel implanted with $2 \times 10^{17} \text{ B}^+/\text{cm}^2$ at 40 keV and $2 \times 10^{17} \text{ Ti}^+/\text{cm}^2$ at 190 keV.

The B KLL lineshape on the surface was indicative of an oxide. The oxide layer was of a thickness normally found on these surfaces, i.e., ~ 5 to 7.5 nm. After 5 minutes of ion milling, the lineshape became boride-like; this lineshape was retained throughout profiling. The carbon and titanium lineshapes did not appear to have been affected by the implantation of boron.

4. CONCLUSIONS

1) A 1:1 ratio of C/Ti over 15 atomic percent is required to produce a durable, wear-resistant surface on 52100 steel.

2) The carbon source does not appear to be consequential. It may come from gettering or implantation.

3) Less wear debris is formed if the energy of the implant is reduced, increasing the concentration of species near the surface, and resulting in lower long-term friction values at the lower doses when Ti is implanted.

4) Order of implantation is insignificant in the Ti + C case.

5) Dual implantation of 2×10^{17} C⁺/cm² at 50 keV and 2×10^{17} Ti⁺/cm² at 190 keV does not render the surface amorphous.

6) Implantation of titanium in conjunction with boron does not improve the long-term friction or wear resistance of 52100 steel.

5. REFERENCES

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